Crystal structure of $C_6H_7ClO_3$ — ban0823

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Abstract

The crystal structure of $C_6H_7ClO_3$ is reported.

Comment

The crystallographic asymmetric unit consists of one $C_6H_7ClO_3$ molecule.

Experimental

The compound was prepared by DMP. The sample ID is 6DP39.

Refinement

The compound is enantiometrically pure. The absolute structure of the crystal has been determined by refinement of the Flack parameter and this establishes the absolute configuration of the molecule.

All H atoms were observed in difference electron density maps prior to their inclusion. They were included at geometrically determined positions and were initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H in the range 0.93–0.98) and U_{iso}(H) (in the range 1.2–1.5 times U_{eq} of the parent atom). Later, the positions of H atoms were refined without restraints.

The largest peaks in the final difference electron density map are located along C—C bonds or near the chlorine atom.

Computing details

Data collection: *COLLECT* (Nonius, 1997-2001).; cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP*II (Johnson 1976) in *TEXSAN* (MSC, 1992-1997); software used to prepare material for publication: *CRYSTALS* (Betteridge *et al.*, 2003).

(ban0823)

Crystal data

$C_6H_7ClO_3$	$V = 665.27\ (2)\ Å^3$
$M_r = 162.57$	$Z = 4$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$
$a = 6.2776\ (1)$ Å	$\mu = 0.51\ mm^{-1}$
$b = 8.4084\ (2)$ Å	$T = 200$ K
$c = 12.6034\ (3)$ Å	$0.33 \times 0.19 \times 0.17$ mm

Data collection

Area diffractometer	1929 independent reflections
Absorption correction: integration via Gaussian method (Coppens, 1970) implemented in maXus (2000)	1823 reflections with $I > 2.0\sigma(I)$
$T_{min} = 0.886$, $T_{max} = 0.922$	$R_{int} = 0.022$
8419 measured reflections	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.024$	Only H-atom coordinates refined
$wR(F^2) = 0.051$	$\Delta\rho_{max} = 0.21\ e\ Å^{-3}$
$S = 0.96$	$\Delta\rho_{min} = -0.19\ e\ Å^{-3}$
1929 reflections	Absolute structure: Flack (1983), 795 Friedel-pairs
113 parameters	Flack parameter: −0.01 (5)

Table 1
Selected geometric parameters (Å, °)

Cl7—C1	1.7468 (13)	C1—C6	1.5031 (18)
O8—C3	1.4346 (16)	C2—C3	1.5013 (18)
O9—C4	1.4455 (15)	C3—C4	1.498 (2)
O9—C5	1.4401 (17)	C4—C5	1.4646 (19)
O10—C6	1.4248 (15)	C5—C6	1.5048 (19)
C1—C2	1.3266 (17)		
C4—O9—C5	61.00 (9)	C3—C4—C5	120.68 (12)
Cl7—C1—C2	119.68 (10)	O9—C4—C5	59.32 (9)
Cl7—C1—C6	114.07 (9)	C4—C5—O9	59.68 (8)
C2—C1—C6	126.23 (12)	C4—C5—C6	122.68 (12)
C1—C2—C3	123.39 (12)	O9—C5—C6	114.15 (11)
C2—C3—O8	110.11 (12)	C5—C6—C1	111.60 (10)
C2—C3—C4	114.01 (11)	C5—C6—O10	106.91 (12)
O8—C3—C4	106.28 (11)	C1—C6—O10	110.39 (11)
C3—C4—O9	114.74 (12)		

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supplementary materials

Crystal structure of $C_6H_7ClO_3$ — ban0823

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Crystal data

$C_6H_7ClO_3$	D_x = 1.623 Mg m^{-3}
M_r = 162.57	Mo $K\alpha$ radiation λ = 0.71073 Å
Orthorhombic, $P2_12_12_1$	Cell parameters from 6639 reflections
a = 6.2776 (1) Å	θ = 2.6–30º
b = 8.4084 (2) Å	μ = 0.51 mm^{-1}
c = 12.6034 (3) Å	T = 200 K
V = 665.27 (2) Å^3	Plate, colourless
Z = 4	0.33 × 0.19 × 0.17 mm
F_{000} = 336	

Data collection

Area diffractometer	1823 reflections with $I > 2.0\sigma(I)$
Monochromator: graphite	R_{int} = 0.022
T = 200 K	θ_{max} = 30.0º
φ and ω scans with CCD	θ_{min} = 2.9º
Absorption correction: integration via Gaussian method (Coppens, 1970) implemented in maXus (2000)	h = −8→8
T_{min} = 0.886, T_{max} = 0.922	k = −9→11
8419 measured reflections	l = −17→16
1929 independent reflections	

Refinement

Refinement on F^2	Only H-atom coordinates refined
Least-squares matrix: full	Method, part 1, Chebychev polynomial, (Watkin, 1994, *P*rince, 1982) [*w*eight] = 1.0/[A_0*T_0(x) + A_1*T_1(x) ··· + A_{n-1}]*T_{n-1}(x)] where A_i are the Chebychev coefficients listed below and x = *F* /*F*max Method = Robust Weighting (*P*rince, 1982) W = [*w*eight] * [1-(delta*F*/6*sigma*F*)^2]^2 A_i are: 27.9 40.0 18.4 3.83
$R[F^2 > 2\sigma(F^2)]$ = 0.024	$(\Delta/\sigma)_{max}$ = 0.004
$wR(F^2)$ = 0.051	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
S = 0.96	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
1929 reflections	Extinction correction: None

113 parameters	Absolute structure: Flack (1983), 795 Friedel-pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: −0.01 (5)
Hydrogen site location: inferred from neighbouring sites	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl7	0.37240 (6)	0.55512 (5)	0.51521 (3)	0.0381
O8	0.52846 (19)	0.67451 (14)	0.13888 (8)	0.0366
O9	0.82680 (16)	0.46367 (12)	0.24097 (8)	0.0332
O10	0.76161 (19)	0.36440 (12)	0.45470 (9)	0.0357
C1	0.5505 (2)	0.59428 (14)	0.41199 (10)	0.0258
C2	0.4864 (2)	0.67800 (15)	0.32869 (10)	0.0263
C3	0.6287 (2)	0.71864 (15)	0.23686 (10)	0.0284
C4	0.8395 (2)	0.63528 (16)	0.23876 (11)	0.0313
C5	0.9076 (2)	0.54615 (18)	0.33266 (11)	0.0319
C6	0.7708 (2)	0.52964 (15)	0.43007 (11)	0.0284
H21	0.342 (3)	0.7146 (19)	0.3251 (13)	0.0319*
H31	0.658 (3)	0.838 (2)	0.2389 (13)	0.0350*
H41	0.946 (3)	0.671 (2)	0.1889 (14)	0.0381*
H51	1.064 (3)	0.530 (2)	0.3423 (13)	0.0383*
H61	0.848 (3)	0.5940 (19)	0.4910 (12)	0.0344*
H81	0.449 (4)	0.739 (2)	0.1211 (17)	0.0558*
H101	0.809 (4)	0.348 (3)	0.5127 (17)	0.0535*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl7	0.04207 (17)	0.04480 (18)	0.02735 (14)	0.00543 (16)	0.00505 (13)	0.00561 (14)
O8	0.0466 (6)	0.0393 (5)	0.0238 (4)	0.0114 (5)	−0.0042 (4)	0.0002 (4)
O9	0.0376 (5)	0.0267 (4)	0.0354 (5)	0.0030 (4)	0.0008 (4)	−0.0046 (4)
O10	0.0432 (6)	0.0264 (5)	0.0376 (5)	0.0013 (4)	−0.0150 (5)	0.0055 (4)
C1	0.0294 (6)	0.0248 (5)	0.0231 (5)	0.0004 (4)	−0.0021 (5)	−0.0019 (4)
C2	0.0285 (6)	0.0245 (5)	0.0258 (5)	0.0024 (5)	−0.0014 (5)	−0.0007 (5)
C3	0.0349 (6)	0.0244 (5)	0.0259 (5)	0.0008 (5)	−0.0005 (5)	0.0005 (4)
C4	0.0309 (7)	0.0278 (6)	0.0350 (6)	−0.0034 (5)	0.0043 (6)	0.0006 (5)
C5	0.0257 (6)	0.0299 (6)	0.0402 (7)	−0.0002 (5)	−0.0038 (5)	−0.0017 (6)
C6	0.0294 (6)	0.0247 (6)	0.0311 (6)	−0.0001 (5)	−0.0085 (5)	−0.0003 (5)

Geometric parameters (Å, °)

Cl7—C1	1.7468 (13)	C2—C3	1.5013 (18)
O8—C3	1.4346 (16)	C2—H21	0.957 (18)
O8—H81	0.77 (2)	C3—C4	1.498 (2)
O9—C4	1.4455 (15)	C3—H31	1.022 (17)
O9—C5	1.4401 (17)	C4—C5	1.4646 (19)
O10—C6	1.4248 (15)	C4—H41	0.966 (18)

O10—H101	0.80 (2)	C5—C6	1.5048 (19)
C1—C2	1.3266 (17)	C5—H51	0.997 (17)
C1—C6	1.5031 (18)	C6—H61	1.056 (17)
Cl7···O9^{i}	3.419 (1)	O8···C6^{iii}	3.385 (2)
Cl7···O8^{ii}	3.534 (1)	O9···C2^{v}	3.226 (2)
O8···O10^{iii}	2.689 (2)	O9···C4^{vi}	3.475 (2)
O8···O10^{iv}	2.694 (2)	O9···C3^{v}	3.535 (2)
C3—O8—H81	110.6 (16)	O9—C4—C5	59.32 (9)
C4—O9—C5	61.00 (9)	C3—C4—H41	117.4 (11)
C6—O10—H101	110.5 (15)	O9—C4—H41	111.0 (11)
Cl7—C1—C2	119.68 (10)	C5—C4—H41	118.7 (10)
Cl7—C1—C6	114.07 (9)	C4—C5—O9	59.68 (8)
C2—C1—C6	126.23 (12)	C4—C5—C6	122.68 (12)
C1—C2—C3	123.39 (12)	O9—C5—C6	114.15 (11)
C1—C2—H21	119.7 (10)	C4—C5—H51	117.2 (10)
C3—C2—H21	116.9 (10)	O9—C5—H51	112.1 (10)
C2—C3—O8	110.11 (12)	C6—C5—H51	116.7 (9)
C2—C3—C4	114.01 (11)	C5—C6—C1	111.60 (10)
O8—C3—C4	106.28 (11)	C5—C6—O10	106.91 (12)
C2—C3—H31	108.2 (9)	C1—C6—O10	110.39 (11)
O8—C3—H31	110.9 (9)	C5—C6—H61	106.5 (9)
C4—C3—H31	107.3 (10)	C1—C6—H61	110.3 (9)
C3—C4—O9	114.74 (12)	O10—C6—H61	111.1 (8)
C3—C4—C5	120.68 (12)		
Cl7—C1—C2—C3	179.4 (1)	O10—C6—C1—C2	−128.6 (1)
Cl7—C1—C6—O10	53.1 (1)	O10—C6—C5—C4	128.9 (1)
Cl7—C1—C6—C5	171.82 (9)	C1—C2—C3—C4	9.2 (2)
O8—C3—C2—C1	128.5 (1)	C1—C6—C5—C4	8.2 (2)
O8—C3—C4—O9	−63.9 (1)	C2—C1—C6—C5	−9.9 (2)
O8—C3—C4—C5	−131.6 (1)	C2—C3—C4—C5	−10.1 (2)
O9—C4—C3—C2	57.5 (1)	C3—C2—C1—C6	1.2 (2)
O9—C4—C5—C6	−100.8 (1)	C3—C4—O9—C5	−112.3 (1)
O9—C5—C4—C3	102.4 (1)	C3—C4—C5—C6	1.5 (2)
O9—C5—C6—O10	60.6 (1)	C4—O9—C5—C6	115.0 (1)
O9—C5—C6—C1	−60.2 (1)		

Symmetry codes: (i) −*x*+3/2, −*y*+1, *z*+1/2; (ii) −*x*+1/2, −*y*+1, *z*+1/2; (iii) −*x*+3/2, −*y*+1, *z*−1/2; (iv) −*x*+1, *y*+1/2, −*z*+1/2; (v) −*x*+1, *y*−1/2, −*z*+1/2; (vi) −*x*+2, *y*−1/2, −*z*+1/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O8—H81···O10^{iv}	0.77 (2)	1.94 (2)	2.694 (2)	165 (2)
O10—H101···O8^{i}	0.80 (2)	1.90 (2)	2.689 (2)	169 (2)

Symmetry codes: (iv) −*x*+1, *y*+1/2, −*z*+1/2; (i) −*x*+3/2, −*y*+1, *z*+1/2.